



# **Environmental Protection Department**

## **Waste Certification Program**

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# **Waste Certification Program**

## **Sampling and Analysis Plan**

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## **1. PROJECT DESCRIPTION**

### **1.1 Waste Description**

This sampling and analysis plan (SAP) is generic for all low-level radioactive waste (LLW) streams generated at the Lawrence Livermore National Laboratory (LLNL). Three important types of waste encompassed by this plan are environmental media such as soils and gravel, HEPA filters, and solidified wastes. However, this plan is not limited to these types of waste. The specific details of the waste description for each sampling event are provided on a Sampling and Analysis Worksheet (SAW; see Fig. 1 for an example). Contaminants in the waste will be identified through process knowledge and by sampling and analysis to demonstrate compliance with *Nevada Test Site Defense Waste Acceptance Criteria, Certification, and Transfer Requirements*, NVO-325 (latest version).

### **1.2 Site Background**

LLNL is a research and development facility located in Livermore, California. One of the world's leading scientific research centers, LLNL is operated by the University of California for the Department of Energy. The main site is approximately one square mile, which is bounded by Vasco Road on the west, East Avenue on the south, Greenville Road on the east, and Patterson Pass Road on the north.

After a long history of nuclear weapons design and development, LLNL now focuses on stewardship of the nuclear stockpile, development of measures against nuclear proliferation, and the safe dismantling of a significant fraction of the strategic arsenal. Some of the other important programs at the main site include U-AVLIS, which uses lasers to produce enriched uranium, and Laser Inertial Confinement Fusion, which seeks to harness fusion, which is the energy source of the stars. Facilities that generate LLW at the main site include the Plutonium and Tritium Facilities and Biology and Biotechnology Research (BBR). In addition, there are important waste treatment and environmental remediation activities. However, the LLW covered by this plan is not limited to that generated from any of these examples, but may come from any source within LLNL. Because the research at LLNL is so varied in scope, the waste may come from virtually any type of chemistry and may potentially involve a huge variety of materials.

In addition to the main site, LLNL also maintains Site 300, which is located in the eastern Altamont Hills about 15 miles southeast of Livermore. One of the missions of Site 300 is diagnostic and materials testing of explosive compounds and devices. The two major types of testing are hydrodynamics experiments and advanced conventional weapons testing. Tests in which explosive compounds are detonated are performed on firing tables constructed adjacent to specifically designed control and equipment bunkers.

## **2. PROJECT ORGANIZATION AND RESPONSIBILITIES**

Several groups will be involved in the performance and review of sampling and analysis of LLW under this plan. The Department of Energy, Nevada Operations Office (DOE/NV), will review this plan and concur that it will provide adequate characterization and representative sampling. The Environmental Protection Department's Hazardous Waste Management Division has the responsibility for sampling and submitting the samples to the Chemistry and Materials Science Department Environmental Services (CES) Sample Management Group, which will provide sample and data management, including shipment of the samples to the outside laboratory for analysis. The Waste Certification Program validates the data after it has been received from CES and is responsible for ensuring that all the criteria described in NVO-325 (latest version) are met prior to certifying the waste for final disposal. Specific individuals with responsibilities for each sampling event will be identified on the SAW. The name and address of the outside laboratory chosen for each sampling event will also be listed on the SAW.

## **3. COMPLIANCE WITH NEVADA TEST SITE WASTE ACCEPTANCE CRITERIA**

The criteria set forth in *Nevada Test Site Defense Waste Acceptance Criteria, Certification, and Transfer Requirements*, NVO-325 (latest version), that are relevant to this SAP and the methods for assuring compliance with these criteria are discussed in this section.

### **3.1 Transuranics**

Waste will be assessed for its radiological content. If it has the potential to contain transuranic nuclides, it will be analyzed by gamma and alpha spectroscopy. This analysis will be used to confirm that the waste has a transuranic nuclide concentration less than 100 nCi/g.

### **3.2 Hazardous Waste Components**

LLW to be disposed of at the Nevada Test Site (NTS) cannot exhibit characteristics of, or be listed as, a hazardous waste, as identified in Title 40, Code of Federal Regulations (CFR), Part 261 (RCRA), or under Title 22, California Code of Regulations (CCR). Therefore, prior to disposal at NTS, analytical data must be provided for the waste to assure that no hazardous characteristics under these regulations are present.

Waste will be assessed for its potential to contain hazardous materials. When necessary, appropriate analytical methods will be used to demonstrate that no hazardous materials are present above the applicable regulatory limits and that no listed hazardous waste is present.

### **3.3 Free Liquids**

LLW to be disposed of at NTS cannot contain any free liquids. Minor liquid residue that does not equal or exceed 0.5% by volume of the external container is acceptable. Waste will be assessed by visual inspection. If there is any question that this limit might be exceeded, a Paint Filter Test will be performed.

### **3.4 Particulates**

NVO-325 (latest version) requires demonstration that the waste consists of no more than 1% by weight of particles less than 10  $\mu\text{m}$  in diameter or 15% by weight of particles less than 200  $\mu\text{m}$  in diameter. In most cases, encapsulation or containerization will be used to ensure compliance with this requirement. Sampling or analysis will only be conducted to verify compliance with this criterion when encapsulation and/or containerization are not used.

### **3.5 Gases**

Radioactive gases and compressed gases (e.g., aerosol cans), as defined by 49 CFR 173.115.b, need to be stabilized or absorbed so that the pressure in the waste container does not exceed 1.5 atm at 20°C. This is accomplished through engineering controls when necessary and does not relate to sampling and analysis.

### **3.6 Stabilization**

NVO-325 (latest version) requires that waste be treated to reduce volume and to provide a more physically and chemically stable waste form. When stabilization has been used, sampling and analysis will be done to verify its effectiveness.

### **3.7 Etiologic Agents**

LLW containing etiologic agents, as defined in 49 CFR 173.134, will not be accepted for disposal at NTS. These agents are not present in LLNL's waste, and their absence will be documented in each case on the SAW.

### **3.8 Chelating Agents**

LLW containing chelating agents at concentrations exceeding 1% by weight will not be accepted at NTS. These agents are not present in LLNL's waste at concentrations exceeding this limit, and this will be documented in each case on the SAW.

### **3.9 Polychlorinated Biphenyls (PCBs)**

The NTS requirement for PCBs is that PCB-contaminated LLW will not be accepted for disposal unless the PCB concentration meets municipal solid waste disposal levels of 50 ppm or less. However, NTS has an additional stipulation that it will not accept waste that does not meet disposal

requirements in the state of generation. California regulates PCBs in concentrations greater than 5 ppm; therefore, this more rigorous standard is maintained by NTS for waste coming from California. When PCBs are potentially present in the waste, the appropriate analytical method will be used to verify that they are not above the California limit.

### **3.10 Explosives and Pyrophorics**

LLW containing explosives and/or pyrophoric material in a form that may spontaneously explode or combust if the container is breached will not be accepted at NTS. Specific analyses for the purpose of identifying these types of compounds will be performed when necessary.

### **3.11 Compliance Analysis**

For each sampling event, the Hazardous Materials Waste Certification Engineer (HMWCE) will complete a compliance analysis and document this on the SAW. The analysis will be based on process knowledge and any historical data and will indicate the need for specific analyses to ensure compliance with each of these waste-acceptance criteria.

## **4. QUALITY ASSURANCE (QA) OBJECTIVES**

This section provides information on QA objectives for procedures and for the data. QA considerations for procedures include field and laboratory techniques. Data quality is assessed by determination of the precision, accuracy, representiveness, comparability, and completeness (PARCC) parameters.

### **4.1 QA Objectives for Measurement**

QA is a management system for ensuring that all information, data, and decisions are technically sound and properly documented. All sampling and analysis activities covered by this SAP will be performed in accordance with the QA practices described in this SAP and in related sampling procedures, which will be referenced on the specific SAW. Contract laboratories selected to perform any of the analytical tests will possess State of California Department of Health Services Certification for Hazardous Waste Testing and will conform to all the requirements for qualification described in NVO-325 (latest version).

This SAP contains or references guidance for the following: sampling and decontamination; sample custody; analytical procedures; data reduction, validation, and reporting; internal quality control checks; audits and surveillances; and specific routine procedures used to assess data precision, accuracy, and completeness.

#### 4.1.1 Sampling Quality Control (QC)

Sampling QC is ensured by uniform sample collection, handling, chain-of-custody, and shipping procedures and by evaluation of QC samples collected in the field. Field samples used to assess QC include field blanks, trip blanks, field duplicates, and rinsates. These samples will be collected according to HWM Procedure 400, "Sampling QC," (1) for each sampling event.

#### 4.1.2 Laboratory Procedures and QA/QC

The analytical laboratory will follow the specific procedures requested (see Section 6). In general, these procedures will be from SW-846 (2).

The analytical laboratory must be capable of producing full Contract Laboratory Program (CLP) data packages according to the appropriate EPA Statements of Work. Use of this data-reporting protocol ensures that all necessary laboratory QC samples will be taken. These will include method blanks, laboratory control samples for inorganics, laboratory duplicates, and matrix spikes, as defined in the Statements of Work.

The use of these protocols will ensure that PARCC characteristics are associated with the data from each sampling event.

### 4.2 Audits

In accordance with Procedure WCP-7 "Quality Surveillance," (3), the Waste Certification Program performs regularly scheduled surveillances of the various sampling operations covered by this plan and of the CES Sample Management Group.

Audits of analytical laboratories will be performed on an annual basis by a certified Lead Auditor, according to the requirements of NVO-325 (latest version). Use of the sample audit plates given in *Nevada Test Site Defense Waste Acceptance Criteria, Certification, and Transfer Requirements Laboratory Reference Document*, NVO-325LRD (latest version), is not required. However, if different audit plates are used, the same topics must be covered at approximately the same level of detail.

### 4.3 Sampling Procedures

Procedures to be used in the operations covered by this plan will be the latest authorized versions. The specific procedures for each sampling operation will be listed on the SAW.

### 4.4 Sampler Training

Sampling will be performed only by qualified personnel who are thoroughly familiar with all applicable procedures and who have completed the training course EP5200, "Sampling Hazardous Waste."

#### **4.5 Sample Control**

After the samples have been taken, they will be transferred to the custody of CES using CES Procedure SOP-SM-P510 “Sample Log In” (4) and CES Procedure SOP-CES-P400 “CES Forms” (5). CES will ship them to the outside analytical laboratory using CES Procedure SOP-SM-P511 “Off-site Shipping” (6).

To assure the integrity of each sample, chain-of-custody seals will be affixed to each sample container at the time of sampling. The sampler will make a signed notation on the Chain-of-Custody Form indicating that the seals have been attached.

#### **4.6 Data Validation**

After the analytical data have been received from the laboratory, they will be validated according to the EPA Data Validation Guidelines (7, 8) to verify that all the QC requirements have been met.

### **5. SAMPLING**

#### **5.1 Sampling Objectives**

The objective of the sampling effort is to verify generator knowledge of the waste stream in order to show compliance with NVO-325 (latest version) and to provide legally defensible data for disposal of this waste to NTS.

#### **5.2 Sampling Strategy**

Sampling shall be conducted with consideration to collecting representative samples, minimizing cross-contamination, and reducing exposure to as-low-as-reasonably-achievable (ALARA) levels.

#### **5.3 Sampling Frequency and Selection**

The determination of the frequency of sampling and the selection of the specific containers to be sampled will be done by the HWMCE, in consultation with the HWM Sampling Group Supervisor and the Generator. The HWMCE will document this on the SAW. The person who takes responsibility for the sampling will be identified on the SAW. In many cases, this will be the HWM Sampling Group Supervisor, but other individuals may take this responsibility if they are adequately qualified and trained.

When there are existing historical data, they will be used to calculate the frequency of sampling according to Equation 8 of SW-846 (Ref. 2). If this calculation leads to a number that is less than 10% of the containers in the population, or if there are no historical data, then 10% of the containers will be sampled.

## **5.4 Sampling Methodology**

Sampling will be done according to an approved procedure that will be referenced on the SAW.

## **6. WASTE ANALYSIS SUMMARY**

The waste analysis to be performed shall be conducted in accordance with NVO-325 (latest version) and the methods referenced therein and in accordance with Ref. 2. The specific types of analyses to be performed will be specified by the HMWCE on the SAW, based on the compliance analysis described in Section 3.

When it is necessary to analyze for the presence of potentially hazardous metals, the samples will undergo the Waste Extraction Test (WET) procedure (sometimes referred to as the Soluble Threshold Limit Concentration [STLC] procedure) described in Appendix II of Section 66261 of Title 22. If the concentration of an extracted bioaccumulative or persistent toxic substance equals or exceeds that given as the STLC value, pursuant to Title 22, the waste is hazardous. For inorganics, the WET (STLC) procedure has been chosen by LLNL for its SAPs in preference to the Toxicity Characteristic Leaching Procedure (TCLP) referred to in NVO-325 (Rev. 1) because the WET will give additional metal constituents and is a more rigorous test. Comparison data for these two procedures may be found in Ref. 9, which also contains a list of inorganic and organic persistent and bioaccumulative toxic substances and their STLC and Total Threshold Limit Concentration (TTLC) values. Other typical methods selected include 1311 TCLP Extraction, 8260 Volatiles, 8270 Semivolatiles, 8080 PCBs, and 9095 for the Paint Filter Test. However, other methods may be used when appropriate.

For radiological analysis, methods typically used will include liquid scintillation for tritium analysis, gamma spectroscopy, and alpha spectroscopy when deemed necessary. Alpha spectroscopy will typically involve the uranium, thorium, and plutonium series. However, other methods may be used when appropriate.

## **7. DATA INTERPRETATION**

If none of the resulting hazardous analyte concentrations equals or exceeds the regulatory threshold for that substance, the samples taken can be considered to be an adequate demonstration that the waste in the containers as represented by these analytical data are nonhazardous and, therefore, radioactive waste. If one or more concentrations equals or exceeds the regulatory threshold, the question arises whether the contamination is localized with respect to that particular container, localized with respect to all of the containers, or general, thus requiring analysis of each container. After validating the data, the HMWCE will assess what additional sampling, if any, may be required in accordance with the statistical methodology cited in Ref. 2.

This analysis applies to listed hazardous waste as well as to characteristic waste.

In all cases, any containers with hazardous materials above the applicable regulatory limits or with any listed waste will be segregated from the population and not shipped to NTS. If it is possible to reprocess the waste according to an approved procedure, the container may be resampled after reprocessing to determine if the reprocessing has reduced the level of hazardous materials to acceptable levels. If this is the case, and the data can be validated, then the reprocessed waste may be shipped.

## **8. REFERENCES**

1. HWM Procedure 400, "Sampling QC," (latest version).
2. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, (latest edition), U. S. Environmental Protection Agency.
3. WCP Procedure WCP-7, "Quality Surveillance," (latest version).
4. CES Procedure SOP-SM-P510, "Sample Log In," (latest version).
5. CES Procedure SOP-CES-P400, "CES Forms," (latest version).
6. CES Procedure SOP-SM-P511, "Off-Site Shipping," (latest version).
7. *Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analysis*, prepared for the Hazardous Site Evaluation Division, USEPA, by the USEPA Data Review Work Group, July 1, 1988.
8. *National Functional Guidelines for Organic Data Review*, USEPA Contract Laboratory Program, revised June 1991.
9. *Application to Ship Low-Level Radioactive Waste to the Nevada Test Site*, Appendix B, Sub-Appendix A-1, pp. 16-24 and G-2 - G-15, Lawrence Livermore National Laboratory, UCRL-CR-111746 (Rev. 1), 1993.

<b>SAMPLING AND ANALYSIS WORKSHEET</b>																																	
Worksheet Number ____	Page 1 of ____																																
<p><b>Section I. Process Knowledge Information: (To be completed by generator)</b></p> <p>General Information: Attach additional sheets if necessary.</p> <p>1. Waste generation location (Bldg./Room or Area) _____            Current location of waste if different from above _____</p> <p>2. Description of waste (Example: soil, gravel, filters, solidified waste oil):            _____            _____</p> <p>3. Describe process/activity that generated the waste (Examples: test drilling, core sampling):            _____            _____</p> <p><input type="checkbox"/> This waste is from the treatment facility, the process conducted for the final waste form is  <input type="checkbox"/> Solidification, <input type="checkbox"/> Encapsulation, <input type="checkbox"/> Other (explain) _____  <input type="checkbox"/> See applicable waste disposal requisitions for the process that originated the waste.  <input type="checkbox"/> List of original disposal requisitions attached.</p> <p>4. Procedures used when generating the waste: _____</p> <p>5. List and attach any additional supporting information (e.g. existing analytical data, investigative reports):            _____            _____</p> <p>6. Program/organization that generated the waste: _____</p> <p>7. Quantity of waste: Weight _____ No. of Drums _____ No. of Boxes _____</p> <p>8. Container type (if already packaged): <input type="checkbox"/> 55 gallon drum, <input type="checkbox"/> 30 gallon drum, <input type="checkbox"/> 4x4x7 box, <input type="checkbox"/> 2x4x7 box  <input type="checkbox"/> Other _____</p> <p><b>Waste Evaluation:</b></p> <p>9. Does the waste contain any of the following:            Verified by: <del>VI=Visual Inspection</del>: <del>PK=Process Knowledge</del></p> <table style="width: 100%; border: none;"> <tr> <td style="width: 40%;">a. Grease/oil</td> <td><input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Unknown <input type="checkbox"/> VI <input type="checkbox"/> PK</td> </tr> <tr> <td>b. Hazardous residues</td> <td><input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Unknown <input type="checkbox"/> VI <input type="checkbox"/> PK</td> </tr> <tr> <td colspan="2">If yes, what are the residues _____</td> </tr> <tr> <td>c. Entrapped liquids</td> <td><input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Unknown <input type="checkbox"/> VI <input type="checkbox"/> PK</td> </tr> <tr> <td colspan="2">If yes, is it less than 0.5% by volume of the waste? <input type="checkbox"/> Yes <input type="checkbox"/> No</td> </tr> <tr> <td colspan="2">What is the liquid? _____</td> </tr> <tr> <td>d. Particulates [<math>&gt; 1\%</math> by weight of <math>&lt; 10</math>-micrometer diameter (flour) or <math>&gt; 15\%</math> by weight of <math>&lt; 200</math>-micrometer diameter (sand)]</td> <td><input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Unknown <input type="checkbox"/> VI <input type="checkbox"/> PK</td> </tr> <tr> <td>e. Compressed gases</td> <td><input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Unknown <input type="checkbox"/> VI <input type="checkbox"/> PK</td> </tr> <tr> <td>f. Etiologic agents</td> <td><input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Unknown <input type="checkbox"/> VI <input type="checkbox"/> PK</td> </tr> <tr> <td>g. Chelating agents</td> <td><input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Unknown <input type="checkbox"/> VI <input type="checkbox"/> PK</td> </tr> <tr> <td colspan="2">If yes, is the concentration less than 1% by weight? <input type="checkbox"/> Yes <input type="checkbox"/> No</td> </tr> <tr> <td>h. PCBs (capacitors, etc.)</td> <td><input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Unknown <input type="checkbox"/> VI <input type="checkbox"/> PK</td> </tr> <tr> <td>i. Explosives</td> <td><input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Unknown <input type="checkbox"/> VI <input type="checkbox"/> PK</td> </tr> <tr> <td>j. Pyrophorics</td> <td><input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Unknown <input type="checkbox"/> VI <input type="checkbox"/> PK</td> </tr> <tr> <td>k. 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WCP 0098 (9/18/96)

Figure 1

## SAMPLING AND ANALYSIS WORKSHEET

Worksheet Number \_\_\_\_

Page 2 of \_\_\_\_

**Section I. continued**

## Radiological Characterization

10. Radionuclides present in the waste and the estimated activity for each nuclide:
- ☐
- Unknown

Radionuclide	Activity (Ci)	Radionuclide	Activity (Ci)
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

☐ See attached sheet    ☐ The above and ☐ attached nuclides are the suspect nuclides, activity unknown.

11. Determination of radionuclides:
- ☐
- Unknown

☐ Process Knowledge: Explain: (Example: Inventory Controls) \_\_\_\_\_

12. Determination of Activity:
- ☐
- Unknown

☐ Gamma Spectroscopy☐ Mass Balance☐ High Sensitivity Neutron Instrument☐ ISAM Method☐ Liquid Scintillation☐ Alpha Spectroscopy☐ Mass-to-Curie Conversion☐ Tritium Off-Gas Measurement☐ Other (explain) \_\_\_\_\_

List procedure(s) followed: \_\_\_\_\_

☐ DPM or CPM to Curie Survey: Instrument \_\_\_\_\_

Probe \_\_\_\_\_

Attach memo describing methodology used.

Generator (please print) \_\_\_\_\_

Extension \_\_\_\_\_

Signature \_\_\_\_\_

Date \_\_\_\_\_

WCP 0098 (9/18/96)

Figure 1, continued

**SAMPLING AND ANALYSIS WORKSHEET**

Worksheet Number \_\_\_\_

Page 3 of \_\_\_\_

**Section II. Sampling (To be completed by Hazardous Materials WCE)**

1. Person/organization responsible for sampling: \_\_\_\_\_  
Responsible person has been trained. ☐ Yes ☐ No

2. Analytical Methods

Check all methods that the waste must be analyzed by:

- |   |   |                                       |
|---|---|---------------------------------------|
| <input type="checkbox"/> Volatiles 8260 (TCLP ZHE 1311) | <input type="checkbox"/> Gross alpha beta   | <input type="checkbox"/> Other: _____ |
| <input type="checkbox"/> Semivolatiles 8270             | <input type="checkbox"/> Alpha Spectroscopy | <input type="checkbox"/> _____        |
| <input type="checkbox"/> STLC Metals (including Hg)     | <input type="checkbox"/> Gamma Spectroscopy | <input type="checkbox"/> _____        |
| <input type="checkbox"/> PCBs 8080                      | <input type="checkbox"/> Tritium            | <input type="checkbox"/> _____        |

Name and address of Laboratory to be used: \_\_\_\_\_

Completed by: Print \_\_\_\_\_

Signature \_\_\_\_\_

Date \_\_\_\_\_

**Section III. Sampling Frequency and Selection (To be completed by Hazardous Materials WCE)**

Completed by: Print \_\_\_\_\_

Signature \_\_\_\_\_

Date \_\_\_\_\_

**Section IV. Sampling Strategy (To be completed by Hazardous Materials WCE)**

Completed by: Print \_\_\_\_\_

Signature \_\_\_\_\_

Date \_\_\_\_\_

**Section V. Sampling Methodology (To be completed by Hazardous Materials WCE)**

Completed by: Print \_\_\_\_\_

Signature \_\_\_\_\_

Date \_\_\_\_\_

WCP 0098 (9/18/96)

**Figure 1, continued**